[40 FR 7049, Feb. 18, 1975, as amended at 41 FR 52693, Dec. 12, 1976; 44 FR 37917, June 29, 1979; 52 FR 24728, July 1, 1987]

§ 53.33 Test procedure for methods for lead.

(a) *Sample collection.* Collect simultaneous 24-hour samples (filters) of lead at the test site or sites with both the reference and candidate methods until at least 10 filter pairs have been obtained. If the conditions of § 53.30(d)(4) apply, collect at least 10 common samples (filters) in accordance with § 53.30(d)(4) and divide each to form the filter pairs.

(b) *Audit samples.* Three audit samples must be obtained from the Director, Quality Assurance Division (MD–77), Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Research Triangle Park, NC 27711. The audit samples are ¾ × 8-inch glass fiber strips containing known amounts of lead at the following nominal levels: 100 µg/strip; 300 µg/strip; 750 µg/strip. The true amount of lead in total µg/strip will be provided with each audit sample.

(c) *Filter analysis.* (1) For both the reference method and the audit samples, analyze each filter extract 3 times in accordance with the reference method analytical procedure. The analysis of replicates should not be performed sequentially (i.e., and single sample should not be analyzed three times in sequence). Calculate the indicated lead concentratons for the reference method samples in µg/m³ for each analysis of each filter. Calculate the indicated total lead amount for the audit samples in µg/strip for each analysis of each strip. Label these test results as R_{1A}, R_{1B}, R_{1C}, R_{2A}, R_{2B}, . . ., Q_{1A}, Q_{1B}, $_{1C}$, ..., where R denotes results from the reference method samples; Q denotes results from the audit samples; 1, 2, 3 indicates filter number and A, B, C indicates the first, second, and third analysis of each filter, respectively.

(2) For the candidate method samples, analyze each sample filter or filter extract three times and calculate, in accordance with the candidate method, the indicated lead concentration in µg/m³ for each analysis of each filter. Label these test results as C_{1A}, C_{1B}, C_{2C}, . . ., where C denotes results from the candidate method. (For candidate methods which provide a direct measurement of lead concentrates without a separable procedure, $C_{1A}=C_{1B}=C_{1C}$, $C_{2A}=C_{2B}=C_{2C}$, etc.)

(d) For the reference method, calculate the average lead concentration for each filter by averaging the concentrations calculated from the three analyses:

$$R_{i\,ave} = \frac{R_{iA}+R_{iB}+R_{iC}}{3},$$

where i is the filter number.

(e) Disregard all filter pairs for which the lead concentration as determined in the previous paragraph (d) by the average of the three reference method determinations, falls outside the range of 0.5 to 4.0 µg/m³. All remaining filter pairs must be subjected to both of the following tests for precision and comparability. At least five filter pairs must be within the 0.5 to 4.0 µg/m³ range for the tests to be valid.

(f) *Test for precision.* (1) Calculate the precision (P) of the analysis (in percent) for each filter and for each method, as the maximum minus the minimum divided by the average of the three concentration values, as follows:

$$P_{Ri} = \frac{R_{i\,max} - R_{i\,min}}{R_{i\,ave}} \times 100\%,$$

or

$$P_{Ci} = \frac{C_{i\,max} - C_{i\,min}}{C_{i\,ave}} \times 100\%,$$

where i indicates the filter number.

(2) If any reference method precision value (P_{Ri}) exceeds 15 percent, the precision of the reference method analytical procedure is out of control. Corrective action must be taken to determine the source(s) of imprecision and the reference method determinations must be repeated according to paragraph (c) of this section, or the entire test procedure (starting with paragraph (a)) must be repeated.

(3) If any candidate method precision value (P_{Ci}) exceeds 15 percent, the candidate method fails the precision test.

(4) The candidate method passes this test if all precision values (i.e., all

P_{Ri}'s and all P_{Ci}'s) are less than 15 percent.

(g) *Test for accuracy.* (1) For the audit samples calculate the average lead concentration for each strip by averaging the concentrations calculated from the three analyses:

$$Q_{i\,ave} = \frac{Q_{iA}+Q_{iB}+Q_{iC}}{3},$$

where i is audit sample number.

Calculate the percent difference (D_q) between the indicated lead concentration for each audit sample and the true lead concentration (T_q) as follows:

$$D_{qi} = \frac{Q_{i\,ave} - T_{qi}}{T_{qi}} \times 100$$

(2) If any difference value (D_{qi}) exceeds ±5 percent the accuracy of the reference method analytical procedure is out of control. Corrective action must be taken to determine the source of the error(s) (e.g., calibration standard discrepancies, extraction problems, etc.) and the reference method and audit sample determinations must be repeated according to paragraph (c) of this section or the entire test precedure (starting with paragraph (a)) must be repeated.

(h) *Test for comparability.* (1) For each filter pair, calculate all nine possible percent differences (D) between the reference and candidate methods, using all nine possible combinations of the three determinations (A, B, and C) for each method, as:

$$D_{in} = \frac{C_{ij} - R_{ik}}{R_{ik}} \times 100\%,$$

where

i is the filter number, and n

numbers from 1 to 9 for the nine possible difference combinations for the three determinations for each method (j= A, B, C, candidate; k= A, B, C, reference).

(2) If none of the percent differences (D) exceed ±20 percent, the candidate method passes the test for comparability.

(3) If one or more of the percent differences (D) exceed ±20 percent, the candidate method fails the test for comparability.

(i) The candidate method must pass both the precision test and the comparability test to qualify for designation as an equivalent method.

TABLE C–3—TEST SPECIFICATIONS FOR LEAD METHODS

Concentration range, µg/m³	0.5–4.0
Minimum number of 24-hr measurements	5
Maximum analytical precision, percent	15
Maximum analytical accuracy, percent	±5
Maximum difference, percent of reference method	±20

[44 FR 37917, June 29, 1979, as amended at 52 FR 24728, July 1, 1987]

§ 53.34 Test procedure for methods for PM_{10}.

(a) *Sample collection.* Using three reference method samplers collocated with three candidate method samplers, collect a minimum of 15 sets of simultaneous 24-hour PM_{10} samples at each of two test sites (i.e., a minimum of 30 sets of samples, each consisting of three reference method and three candidate method samples collected simultaneously, 180 samples total). If the conditions of § 53.30(d)(4) apply, collect sample sets only with the three reference method samplers.

(b) *Sample analysis.* Analyze each sample (or the same sample if § 53.30(d)(4) applies) according to the reference method or candidate method, as appropriate, and determine the PM_{10} concentration in µg/m³.

(c) *Test for comparability.* (1) For each of the sample sets, calculate the average PM_{10} concentration obtained with the reference method samplers:

$$\overline{R}_j = \frac{\sum_{i=1}^{3} R_{ij}}{3}$$

where R denotes results from the reference method, i is the sampler number, and j is the set.